Reduction kinetics of iron ore pellets made by addition of -100# size fines in conventional pellets

A THESIS SUBMITTED IN PARTIAL FULLFILLMENT OF

THE REQUIREMENTS FOR THE DEGREE OF

Bachelor of Technology

In

Metallurgical and Materials Engineering

By

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CERTIFICATE

This is to certify that the thesis entitled "**Reduction kinetics of iron ore pellets made by addition of -100# size fines in conventional pellets** "submitted by **Mr. Satwinder Singh Kalsi** & **Mr. Avinash Kumar** in partial fulfillment of the requirements for the degree of Bachelor of Technology in Metallurgy and Materials engineering at National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by them under my supervision and guidance. To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other university/institute for the award of any degree or diploma.

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ABSTRACT

The increasing trend in the prices of steel scrap and its short supply led the steel technologists to find a suitable charge mix in the form of sponge iron or Direct Reduced Iron (DRI).

Sponge Iron or DRI is obtained from the direct reduction of iron ore and has iron content between 84 to 95 percent. Technically and technologically, sponge iron has been found to be a suitable material for charging in blast furnaces. In the existing blast furnace an increase in production by at least 25 to 35% can be achieved by using pre reduced iron ore. Sponge iron is a better substitute for scrap for steelmaking through EAF / IF routes due to its homogenous nature, Improved productivity and lower coke consumption. Also majority of fines which are generated during the course of handling, mining, transportation etc are exported at a through away price which need to be utilized by making iron ore pellets for sponge iron making. According to industry experts, the preference for usage of DRI will lead to the use of 80 percent DRI in the charge mix in EAFs by 2009-10 which may even reach the 85 percent level by 2011-12 in the various regions of India.

A lot of investigations have been carried out on direct reduction process of iron oxides by carbonaceous materials, but little work has been done on the characterization of properties and reduction behavior of iron ore of some mines of Orissa. In the present project work, an attempt has been made to study the reduction behavior and kinetics of fired iron ore pellets. The effect of different reduction parameters such as temperature (900-950°C), time (15-60 min.), reductant quality, mixing of particles of different sizes at different ratios for pellet preparation etc. on the reduction, swelling behavior of iron ore pellets. These form the subject matter of the thesis.

First chapter gives the introduction about the subject. It speaks about the need of DRI industry, scope, present status and future planning of DRI industry in India, world wise DRI production, etc. It gives a general idea of raw materials quality required for producing DRI, especially in Rotary Kiln. The second chapter deals with the literature survey.

The third chapter deals with planning of experiments ,selection of raw materials, preparation of samples, preparation of iron ore pellets, experimental procedure , evaluation of reduction.

The results obtained and the discussions made from these observations have been outlined in chapter four. The result for fired iron ore pellets indicated an increase in degree of reduction with increase in reduction temperature (900-950ºC) and time. The reduction kinetics of Sakaruddin iron ore pellets were studied in temperature range of 900-950ºC. Analysis of swelling behavior was also done. Results obtained from chapter four have been summarized in chapter five. Lastly, these conclusions have been followed by the list of references.

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CHAPTER 1

INTRODUCTION

1. INTRODUCTION

Globally, iron ore is the major feedstock for BF-BOF steelmaking route which produced 65.4 percent of world crude steel in 2005. But the process requires various types of treatment of raw materials, involving high capital costs and substantial investment on infrastructure. It also leads to environmental problems and has a long gestation period.

To find a way out of this shortcoming of BF-BOF process, EAF steelmaking was introduced long ago. The share of EAF steelmaking in the global output of crude steel has increased substantially from 26.6 percent in 1988 to 33.1 percent in 2005. The increasing trend in the prices of steel scrap and its short supply led the steel technologists to find a suitable charge mix in the form of sponge iron or Direct Reduced Iron (DRI).

The growth of DRI industry took place in its nascent form the latter half of the 20th century, until then steel scrap constituted the major part of our import, next to petroleum product. Then Indian government imposed some curtailment measure and search for the alternatives began, and direct reduced iron was resulted as an alternative to the steel scrap, which is produced by the direct reduction of iron ore in the form of lump or pellet. In direct reduction (DRI) process ,there a good flexibility of using different kind of reductants like lower grade non coking coal, char coal, natural gas etc. The fast depletion of high grade cooking coal, reserves restricts the use of coke in conventional blast furnace-oxygen steel making route, in India.

Being enriched with good quality Iron ore along with vast reserves of non coking Coal, which likely last for another 200 years or so India is in an adventitious for coal based Sponge Iron plants. The total gross reserves of coking and non coking coal in India are approximately 11,602 and 71,400 million tons respectively. From this prospective, the rotary kiln (coal based) DR process have developed well and vigorously in the country instead of natural gas based Shaft furnace or Retort furnace.

In order to accept the potential of the fact, it is rather imperative to understand the basic mechanism involved in DR process using non coking coal as reductant.

The reduction of iron ore by carbon is one of the most important reactions in iron making in blast furnace, rotary kiln and electric smelting furnace. Extensive studies has been carried out on the reduction behavior of iron ore mixed with carbon/char/graphite/coke etc , which reveals that the reduction reaction take place via gaseous intermediate like Carbon monoxide and Carbon

dioxide. As such it is evident that, the actual direct reduction doesn't need any gaseous medium to be carried out. But of late it has been rather well accepted that, the reduction of Iron oxide by Carbon in blast furnace and direct reduction process of sponge Iron production is mostly the result of indirect reduction.

Currently a lot of emphasis is being given to direct reduction process because use of pre-reduced pellets or sponge iron as feed for blast furnace, induction furnaces and basic oxygen furnaces, despite some associated drawbacks, offers much scope for improving both productivity and economy in coke consumption.

1. 1 YEAR-WISE DRI PRODUCTION IN INDIA AND IN THE WORLD

A year-wise production of sponge iron in the world including India is given in Table-1. 1.

According to recent analysis, it has been observed that beginning with a meager production of 0.79 million tons in 1970, the world sponge iron production went upto 55.85 million tons in 2005, as shown in table-1.it is clear from the table that the world sponge iron production has been increased nearly 225% from the year 1990 to 2005.

In the year 2002 India became the largest producer of sponge iron in the world with a production of 5.48 million tones and still it has retained its first slot in the world rating of sponge iron production.

Out of 16.27 MT of sponge iron produced in 2006-07 the contribution of coal based sponge iron units is around 11.01 MT and that of has based units are 5.26 MT. This large difference in contribution of G.B.S.I.U. and C.B.S.I.U. is due to scarcity of natural gas and abundant availability of non-coking coal in India.

This phenomenal growth of DRI industries is driven by increasing demand of steel in India and as well as in the world. Now India is the 6th largest steel producer in the world with a production of 42 MT/Annum. And out of this around 45% of steel is coming from the DRI-EAF route.

As such today DR is definitely on a strong ground. Future growth is guaranteed and driven by growth of steel making and insufficient supply of prime scrap especially in terms of quality.

TABLES

Table-1.1

Year wise Sponge Iron Production in World and in India

Year	World scenario		Indian scenario	
	Production (MT)	Growth (%)	Production (MT)	Growth (%)
1990-91	17.68	_	NA	_
1991-92	19.32	9.27	1.31	NA
1992-93	20.51	6.15	1.44	9.92
1993-94	23.65	15.30	2.40	66.66
1994-95	27.37	15.70	3.39	41.25
1995-96	30.67	12.00	4.40	29.79
1996-97	33.30	8.40	5.00	13.63
1997-98	36.19	8.88	5.30	6.00
1998-99	36.96	2.50	5.22	-1.50
1999-00	38.60	4.10	5.34	22.98
2000-01	43.78	11.90	5.48	26.21
2001-02	40.32	-6.99	5.43	-9.12
2002-03	45.08	12.00	6.9	27.07
2003-04	49.45	9.69	8.08	17.10
2004-05	54.60	10.41	10.30	27.45
2005-06	55.85	2.23	11.47	11.35
2006-07	59.8	_	16.27	_
2007-08	68.5	_	20	_

Source: steelworld.com

1. 2 IRON ORE RESERVES IN INDIA

India's global position is fifth in terms of iron ore reserves. It has 25 billion tons of reserves, of Which 15 billion tons are reported to be hematite and rest magnetite at cut off grades of 55% iron as per Indian bureau of mines (IBM). India produces around 155 million tons of iron ore (Including both lumps and fines) out of which about 52 million tons were used by the domestic steel manufacturers.

Table 1.21 Iron Ore Reserves in India

States	Main Ore	Fe Range (%age)	Alumina (%age)	Phos Max (%age)
A-Orissa, Jharkhand	Haematite	62-64	2-4	0.04-0.1
B-Chattisgarh, MP, Maharashtra	Haematite	64-66	1.0-4.0	0.04-0.15
C-Karnataka	Haematite	62-64	2.0-4.0	0.04-0.09
D-Goa	Haematite	60-63	2.0-4.0	0.04-0.07
E-Karnataka	Magnetite	35-45	1.0	-

States	Major Mines / Deposits
A-Orissa, Jharkhand	Chiria, Noamundi, Joda, Kiriburu, Meghataburu, Thakurani, Bolani, Gua, Malangtoli, Gandhamardan, Daitari
B-Chattisgarh, MP, Maharashtra	Bailadila, Dalli,Rajhara, Rowghat, Mahamaya, Aridongri, Surajgarh
C-Karnataka	Donimalai, Ramandurg, Kumaraswamy, NEB Range, Ettinahatti, Tumti, Belagal
D-Goa	N Goa, S Goa, Redi
E-Karnataka	Kudremukh, Bababudan, Kudachadri

Table 1.22

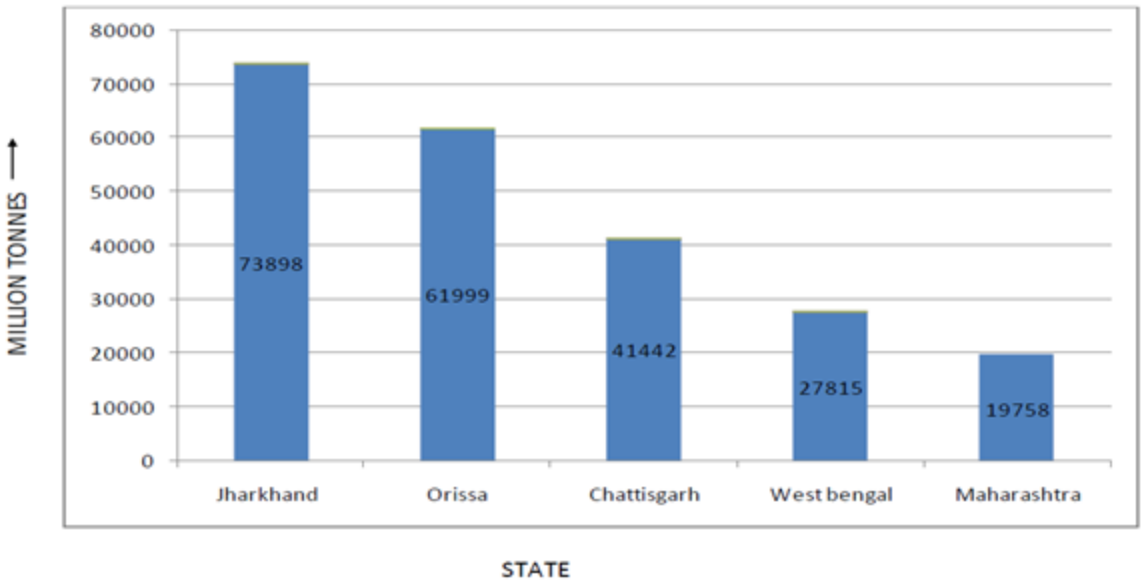
		2000-01	2001-02	2002-03	2003-04	2004-05	2005-06(P)
Crude steel production	(A) Main producers	17254	17762 (2.94)	18982 (6.87)	20012 (5.43)	20015 (0.01)	21694 (8.39)
	(B) Secondary producers	9703	10202 (5.14)	11461 (12.34)	14236 (24.21)	14806 (4.00)	19650 (6.38)
	TOTAL	**26957**	**27964 (3.73)**	**30443 (8.86)**	**34248 (12.50)**	**34821 (1.67)**	**41344 (7.43)**
Iron ore consumption		**36020**	**37713 (4.70)**	**40936 (8.54)**	**44974 (9.86)**	**48150 (7.06)**	**52523 (9.08)**
Iron ore exports		**37270**	**41640 (11.72)**	**48020 (15.32)**	**62570 (30.30)**	**78145 (24.89)**	**89277 (14.25)**
Iron ore production		**80762**	**86226 (6.76)**	**99072 (14.90)**	**122838 (23.99)**	**145942 (18.81)**	**154436 (5.82)**
Surplus iron ore		**7472**	**6873**	**10116**	**15294**	**19647**	**12636**

Source: 1 Joint Plant Committee, Kolkata
2 Indian Bureau of Mines, Nagpur

1.3 COAL RESERVES IN INDIA AND THEIR GRADATION

India is the third largest producer of coal and has the fourth largest coal reserves in the world consisting 10% of the world share of coal reserves. Non coking coal contributes to about 221 billion tons (85%) while coking coal amounts to 32 billion tons (the remaining 15%). Indian coal has high ash content (15- 45%) and low calorific value.

Fig 1.3 Coal state wise distribution



The sponge iron industry is quite comfortable so far the reserves of iron ore and coal are concerned. Total recoverable resources of iron ore in the country account for about 9% of the world's reserves and the major deposits are located in the states of Orissa (34%), Jharkhand (27%), Chhattisgarh (18%), etc. Grade-wise total proved reserves of non-coking coal in India have been outlined in Table 1.31 below.

The gradation of Indian non-coking coals has been carried out on the basis of useful heat value (UHV) and is shown in Table1.32 [1]. As per the present system of coal linkage in India, about 80% of the production of high grade coals (A, B and C) goes to the power sector and the balance 20% to other industrial sectors including sponge iron plants .These data clearly indicate that measures must be taken to utilize D, E, F and G grade coals through better process control.

Table 1.31

Grade-wise proved coal reserves in India

Coal grade	High			Intermediate	Low	Total
	A	B	C	D	E, F & G	
Reserves (million tonne)	961	2,341	7,197	6,216	21,783	38,498

Table 1.32

Gradation of Indian non-coking coals

Grade	Useful heat value (UHV) (Kcal / kg) UHV = 8900 – 138(A+M)	Corresponding (A+M) at 60% RH & 40^0 C	Gross calorific value (GCV) (Kcal / kg) at 5% moisture level
A	> 6200	≤ 19.5	> 6454
B	5601 – 6200	19.6 – 23.8	6050 – 6454
C	4941 – 5600	23.9 – 28.6	5598 – 6049
D	4201 – 4940	28.7 – 34.0	5090 – 5597
E	3361 – 4200	34.1 – 40.0	4325 – 5089
F	2401 – 3360	40.1 – 47.0	3866 – 4324
G	1301 – 2400	47.1 – 55.0	3114 – 3865

A – % Ash ; M – % Moisture

1.4 PRODUCTION, CONSUMPTION, EXPORT AND SURPLUS AVAILABILITY OF IRON ORE LUMPS AND FINES IN INDIA

Some data's of Production, Consumption, Export and Surplus availability of Iron Ore in India, in the year 2003-04, 2004-05, 2005-06 are given in the Table 1.41.

From the Table 1.41 it is quite clear that, the ratios of fines in total export are increasing year by year. About 78.50% in year 2003-04 and has increased to around 84% in the year 2005-06. So it is quite obvious that the fines generated don't find there market in India. After meeting the entire domestic demand and export demand, there was still Iron Ore surplus of 15.30 MT, 19.65 MT, and 12.93 MT, in the year 2003-04, 2004-05 and 2005-06 respectively.

Moreover, around 84% of fines are exported to foreign countries at a throw away price which causes huge economic loss to our countries. If these fines can find their utilization in the country, it would be a great contribution towards our economic growth.

Table 1.41

Year wise production, consumption, export and surplus availability of iron ore lump and fines in India

In Million tons

Year	Iron Ore Production	Domestic Consumption of Iron Ore	Export Of Iron Ore			Surplus Availability of Iron Ore
			Lump Ore	Fines	Total	
2003-04	122.84	44.97	13.45 (21.50)	49.12 (78.50)	62.57 (100)	15.30
2004-05	145.95	48.15	13.54 (17.33)	64.60 (82.67)	78.14 (100)	19.65
2005-06	154.43	52.23	14.30 (16.01)	74.97 (83.99)	89.27 (100)	12.93

(Figures in parenthesis are the percentage of total export)

Source: Indian Bureau of Mines, Nagpur, GMOEA, KIOCL, NMDC, MMTC

1. 5 FUTURE OF DRI-EAF ROUTE OF STEEL MAKING IN INDIA

There exist in the world two main routes for the production of liquid steel: On one side, the "Classical Route" based on blast furnace iron making and conversion of pig iron to steel. On the other side, the "Modern Route" for the production of steel in electric arc furnaces, using as raw material either scrap or direct reduced iron (DRI).The "Classical Route" is based on iron ore reduction in the blast furnace to a high carbon-primary metal (liquid pig iron), followed by the conversion of this hot metal to steel mainly in oxygen converters (in the past open heart furnaces were also used). Although this route continues to produce the major percentage of steel in the world, it requires coal preparation, including coking in coke ovens, as well as ore preparation in sintering or pelletizing plants. Economically, it is mandatory to increase the unit size and the production of each plant, so as to work with larger and more efficient blast furnaces, as well as with higher capacity oxygen converters. In this way, the most economical production capacity of an integrated installation, consisting of a sintering plant, a coke oven battery, a blast furnace and an oxygen converter, is between 2 and 4 Million ton/year of liquid steel.

As opposed to the "Classical Route", the "Modern Route" is based on the melting of metallic iron units in the electric arc furnace. Traditionally, scrap has been used as the main raw material, but the use of direct reduced iron (DRI) is becoming a normal and advantageous practice during melting in the electric arc furnace. During a first stage, iron ores are directly converted to metallic iron in the solid phase, and in a second stage this solid material is converted into liquid steel in the electric arc furnace.

In comparison with the "Classical Route", this technology has the following main advantages:

- Simplicity of the plant operation.
- Flexibility to operate at smaller capacities, down to 300,000 ton/year with an attractive production cost.
- Best environmental impact compared to coking plants and blast furnaces
- New plants can be realized in phases, to optimize the financial structure of the project.
- Possibility of using local energy sources, as this route can operate with natural gas, non cocking coal, petrochemical wastes etc.
- Very attractive investment cost as compared to the blast furnace-BOF route

1.6 RAW MATERIALS FOR DRI AND CRITERIA FOR THEIR SELECTION [2]

- **Iron Ore Fines*:***

In rotary kiln, usually the ore of size between 5 to 18 mm are used. This size range of the ore is produced by crushing and screening the lumpy ore received from the mines. In this process nearly 35% of the ore is produced in the form of fines. For per ton of sponge iron produced, nearly 0.8 tons of iron ore fines are used. The typical sieve analysis of iron ore fines are given below

- -1mm (30- 40%)
- -3mm (35-38%)
- +3,-5mm (28-45%)

Utilization of Iron Ore Fines*:*

The Iron Ore Fines, which are produced as a waste material, can be utilized in the following manner:

- Some quantity of +3 to -5mm can be used along with the charge in rotary kiln by adjusting positive size of coal, etc.
- The iron ore fines can be pelletized and used in the blast furnace for producing pig iron or in rotary kiln for sponge iron making.
- Sinters can be made for iron ore fines and used in the blast furnace.
- Composite pre-reduced pellets (CPR) can be made from iron ore and coal fines for use in iron blast furnace or EAF.
- Sponge iron rods, granules, etc can be made by iron ore fines in coal.

The efficiency and cost effectiveness of DRI production are very sensitive to their raw material characteristics. In a coal based process, these are governed by nature of coal. The following parameters are considered for selection of iron ore for DR process.

- Chemical Composition
- Reducibility
- Physical Characteristics (size, strength etc)

- **Coal Fines:**

The Sponge Iron plants generally use 6mm size coal partially the finer fraction which contains around 32-40% ash is considered as a waste. In Sponge Iron plants around 15-20% fines are generated.

Utilization of Coal Fines:

The Coal fines thus produced can be used in the following manner.

- Generation of Power through fluidized bed combustion boiler.
- For heat hardening of the iron ore pellets produced from waste iron ore fines.
- It can be used in the sinter mixture after beneficiation.

The properties to be considered for selection of coal for DRI production are as follows.

- Proximate Analysis (Fixed Carbon, Volatile matter, Ash, Moisture).
- Sulphur
- Ash fusion temperature
- Ash chemistry

- **Coal Char fines:**

The coal char fines came out of the kiln along with the sponge iron. The generation of the char fines is about 15% of the sponge iron produced. However, the fixed carbon present in the coal char is only 19-21% with 1-2% of volatile matters.

Utilization of coal char fines:

The coal Char produced can be utilized, after suitable treatment in the following areas.

- Preparation of smokeless domestic fuel briquettes along with small amount of coal fines / coke breeze / saw dusts, wood charcoal etc.
- For generation of power, along with coal fines.
- For treating industrial effluents to remove toxic elements.

1.7 AIMS & OBJECTIVES OF THE PROJECT

- Study of the effect of temperature on the degree of reduction of iron ore pellets
- Study of the effect of time on the degree of reduction of iron ore pellets.
- Study of effect of mixing of iron ore particles of different sizes in different ratios on the reduction characteristics of the resulting iron ore pellets.
- Study of effect of time on extent of swelling of iron ore pellets.
- Study of effect of temperature on extent of swelling of iron ore pellets.
- Study of Correlation between Degree of reduction and Percentage swelling of iron ore pellets

CHAPTER-2

LITERATURE SURVEY

2. LITERATURE SURVEY

2.1 DEFINATION OF PELLETS

Pellets are approximately spherical lumps formed by agglomeration of the crushed iron ore fines in presence of moisture and binder, on subsequent induration at 1300°C.

2.2 MECHANISM OF PELLET FORMATION [3]

- Ball Formation – Surface tension of water & gravitational force creates pressure on particles, so they coalesce together & form nuclei which grow in size into ball.
- Induration (Heat Hardening) – Solid state diffusion at particle surfaces at higher temperature cause recrystallisation & growth giving strength

Theory of ball formation

Surface tension of water & gravitational force creates pressure on particles, so they coalesce together & form nuclei which grow in size into ball.

Induration (Heat Hardening)

Solid state diffusion at particle surfaces at higher temperature causes recrystallisation and growth giving strength. The force responsible for the agglomeration of ore fines are capillary action of water and gravitational force of particles. The ball forces occur because of surface tension forces of water and collision between particles. Initially, nuclei of pellet are form from addition of water and the nuclei grow bigger in balls and then into pellets as they pick up loose grain particles during their travel through the drum, which is slightly tilted the angle of tilt determines the time of residence, pellet size and productivity. The size and shape of drum should be such as to obtain the most favorable condition of motion and pressure that is more of rolling as offer to sliding motion. This ensures more effective collision between particles. The capillary action of water under interstitials of the grain causes a contracting effect on then. The pressure of water on the course of ball is sufficiently high so as t compact the constitute grain into a dense mass. The compressive force is directly proportional to the fineness of the grain since the capillary action rises if the decrease in pore radius and the later decreases with increase in fineness. When one particle falls and comes into the gravitational force filled of another particle of which it falls and its neighbors, they adhere because of pressure generated due to gravitational force. Due to this force tremendous amount of pressure is built up which leads to molecular adhesion. The green

pellets obtain are then fired at 1100ºC to ensure complete removal of moisture and then these are heat hardened at 1300ºC or 1 hour in order to ensure the strength of pellets which is due to recrystallisation and grain growth.

2.3 ADVANTAGES OF PELLETS

- ***Good Reducibility***

Because of their high porosity that is (25-30%), pellets usually reduced considerably faster than hard burden sinter or hard natural ores. In the earlier days of palletizing, considerable weight was given to means for securing porosity for higher reducibility.

- ***Good bed Permeability***

Because of their spherical shapes and containing open pores, these are formed very good bed permeability. The shape, size and low angle of repose give minimal segregation and an even charge distribution in the furnace, extending more towards the axis.

- ***High Strength (150-250 kg/cm^2) or more***

Pellets should have sufficient structural strength to with stand, without significant breakage, the normal handling which occurs in the various transportation and handling steps between the pellet furnace an the blast furnace skip.

- ***High Porosity (25-30%)***

Because of high porosity of pellets, these are easily reducible.

- ***Less heat consumption than sintering***
- ***Uniform chemical composition***

The chemical analysis is to degree controllable in the concentration processing within limits dictated by economics.

- ***Easy handling and transportation***
- ***Good resistance to disintegration during furnacing***

It is extremely difficult to determine what happens to pellet after it enters the blast furnace. Questions have arisen as to whether certain type of pellets might disintegrate in the furnace before they reach the smelting zone. Several tests have been advanced to measure this disintegration. One is the Linder test.

- ***Resistance to weathering and freezing***

Because pellets are subjected to extended storage from time of production to time of consumption, they must be resistant to physical breakdown caused by exposure to weather. Moisture and freezing are chief hazards.

Well fired pellets have 20-35% porosity, but pick up only about 3% moisture even after prolonged exposure of all kinds of weather. Freezing and thawing do not result in any significant breakdown. The effect of weathering give some concern if lime or magnesia are used either as an

additives or to flux the pellet. Complete calcinations and chemical combination of these materials with the iron must be attained to prevent hydrolysis on weathering and resultant decrepitation.

2.4 DISADVANTAGES OF PELLETS

- High cost of production.
- Swelling and loss of strength inside the furnace.
- Difficulty of producing fluxed pellets.
- Maximum basicity of the pellets is 1.2

2.5THERMODYNAMICS AND KINETICS OF IRON ORE REDUCTION

CHEMICAL REACTIONS AND THERMODYNAMIC ANALYSIS

Coal contains volatile matter and the iron oxide is Fe_2O_3. On heating, the following reactions take place (Nascimento et al. 1997) :

Coal → char + tar + gases (CO, CO_2, H_2, C_nH_m and N_2) ..... (1)

$Fe_2O_3(s) + 3C(s) \rightarrow 2Fe(s) + 3CO(g)$ ..... (2)

Reduction of hematite :

$Fe_2O_3(s) + CO(g) / H_2(g) \rightarrow 2Fe_3O_4(s) + CO_2(g) / H_2O(g)$ ... (3)

Reduction of magnetite :

$Fe_3O_4(s) + CO(g) / H_2(g) \rightarrow 3FeO(s) + CO_2(g) / H_2O(g)$ ……..…. (4)

Reduction of wustite :

$FeO(s) + CO(g) / H_2(g) \rightarrow Fe(s) + CO_2(g) / H_2O(g)$ … (5)

Besides the above mentioned reactions, the next three possible reactions are :

$C(s) + CO_2(g) \rightarrow 2CO(g)$ ……. (6)

$C(s) + H_2O(g) \rightarrow H_2(g) + CO(g)$.. (7)

Reaction (1) represents thermal decomposition (devolatilization) of coal and it starts in the temperature range of 300 – 500^0C. The products (CO & H_2) of reactions (1) and (2) induce reactions (3), (4) and (5). Carbon gasification reactions with CO_2 and H_2O (6 & 7) occur at temperatures above 800^0C and generates reducing gases (CO & H_2) for the reduction of iron oxides . The gas, in excess to that required for the reactions, flows out from the reactor.

On the basis of thermodynamic analysis from ΔG vs. T and ΔH vs. T plots for the reduction with CO and H_2 done by Komatina & Gudenau 2004 [4], it could be said that the most probable reaction is (3), followed by reactions (4) and (5). The reactions (6) and (7) are highly endothermic and possible at higher temperatures. In comparison to CO gas, the reduction reactions with H_2 gas are mostly endothermic and favoured at higher temperatures.

2.6KINETIC STEPS INVOLVED DURING IRON ORE REDUCTION

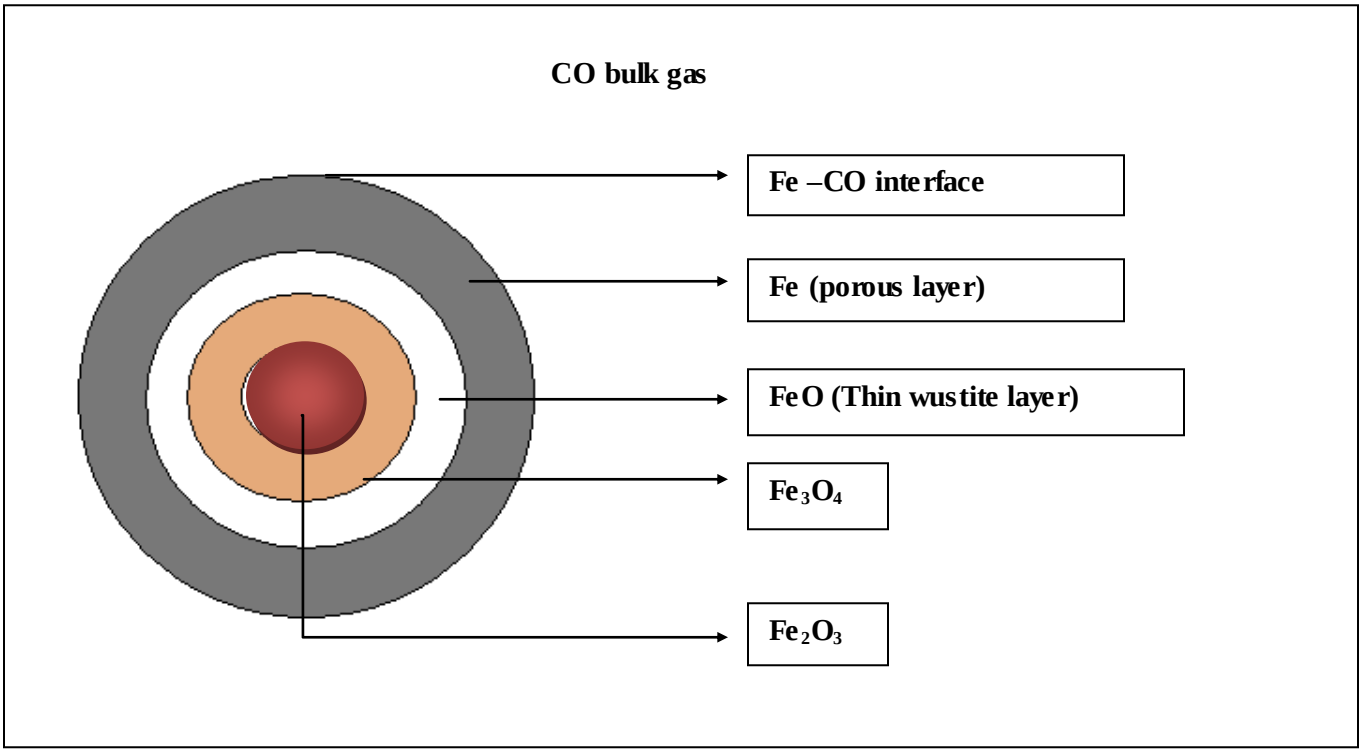


Fig: 2.6 A schematic diagram in modes of reduction of iron oxide

Before the formation of Metallic Layer

The kinetic steps involved in reduction of hematite iron ore by CO/H_2 gas are as follows:

• Transport of CO/H_2 gas from bulk gas phase to Fe_2O_3-CO/H_2interface

• Adsorption of CO/H_2 gas at the Fe_2O_3- CO/H_2interface

• Chemical reaction between $Fe_2O_3$and CO/H_2gas at the Fe_2O_3- CO/H_2interface and desorption of the product gas CO_2/H_2O from this interface.

• Transport of product gas from Fe_2O_3- CO/H_2interface to the bulk gas phase.

After the Formation of Metallic layer

• Transport of CO/H_2 gas from bulk gas phase to the Fe-CO/H_2 interface

• Adsorption of CO/H_2 gas at the Fe- CO/H_2 interface

• Transport of CO/H_2 gas from Fe- CO/H_2 interface to the Fe_2O_3 - Fe interface

• Chemical reaction b/w Fe_2O_3 and CO/H_2 at Fe_2O_3 - Fe interface

• Desorption of the product gas CO_2/H_2O from this interface

• Transport of CO_2/H_2O gas from Fe_2O_3 - Fe interface to Fe-CO/H_2interface

• Transport of the product gas from Fe-CO/H_2 interface to the bulk gas phase

The steps involved are either diffusional or chemical and the slowest of these control the overall rate of reaction.

Rate controlling steps: A reaction may be diffusion/ chemically/ mixed controlled. Several model equations have been proposed to determine the rate controlling step during iron ore reduction. These are as follows:

Kinetic Model Equations Usually Used in Iron Ore Reduction with C/CO/H_2 gas:

Equation	Type of reaction kinetics
$-\ln(1-\alpha) = kt$	Chemical: first order phase boundary reaction control model equation
$1-(1-\alpha)^{1/3} = kt$	Chemical: contracting spherical reaction control model equation
$[1-(1-\alpha)^{1/3}]^2 = kt$	Diffusion: Jander's equation
$1-\frac{2}{3}\alpha-(1-\alpha)^{2/3} = kt$	Diffusion: Ginstling-Brounshtein(GB) diffusion control model equation
$[(1-\alpha)^{-1/3}-1]^2 = kt$	Diffusion: Zhuravlev-Lesokhin-Tempel'man(ZLT) diffusion control model equation

α – Fraction reacted; k – Reaction rate constant; t – time.

Slopes of the lines give the values of reaction rate constants at the concerned temps.. By using Arrhenius equation, the values of activation energy are then determined.

lnK = lnA – E/RT --------A st. line equation

Plots of lnK vs. 1/T give st. lines. Slopes of these st. lines = – E/R. Intercept of these lines with lnK axis give the values of lnA.

Literature indicates that the majority of iron ore reductions are controlled either by C gasification($C+CO_2$) reaction or reduction of FeO. The activation energy values for reduction of iron ores by coal are small compared to the values reported for iron ore-coke and iron ore-graphite systems. This shows that the kinetics of iron ore reduction is highly sensitive to the characteristics of carbon used. Higher reactivity of carbon used gives lower activation energies for reduction.

2.7 FACTORS AFFECTING REDUCTION KINETICS OF IRON ORE:

The following are the factors:

- Physical characteristics of the oxide ore (size, shape, porosity, etc.)
- History of ore preparation (agglomeration, etc.)
- Temperature of the charge
- CO/H_2 content in the gas phase
- Gas flow rate
- Chemical nature of the oxide
- Gangue content in the ore
- Reactivity of the solid fuel
- Pressure applied
- The extent of solid-solid/gas-solid reaction

The investigation on the reduction of iron ore pellets (mixed or composite) with coal fines has been made by a number of workers. T. Sharma studied the effect of Firing Condition and Ingredients on the Swelling Behaviour of lron Ore Pellets [5]. He found that the swelling of iron ore pellet is controlled by the firing temperature, firing time and additives/ingredients present in the pellet. The growth of iron whisker is controlled by these ingredients. The presence of free lime promotes the swelling index of the pellet.

The reduction behavior of hematite and magnetite pellets containing coal char has been studied by Seaton et al[6] , where they have observed higher reduction rate during the initial stage of reduction. This stage comprises the pyrolysis of the remaining volatile matter in char, the

reduction of hematite and magnetite to Wustite and part if Wustite to iron. In fact, they confirmed the presence of the phases by X-ray diffraction analysis. They have indicated that the steps $Fe_2O_3 \rightarrow Fe_3O_4 \rightarrow FeO$ and $Fe_3O_4 \rightarrow FeO$ took place rapidly during early stages of reduction.

The result obtained by Sharma [7] for reduction of iron ore-char mixed pellet has been found to fit in kinetic equation ln (1-f) = kt and both the reactions, i.e. reduction and carbon gasification, have been reported to be coupled in controlling the reaction rate of reactions. The values of activation energies obtained were 166.38 and 160.9 KJ/mole for reduction and carbon gasification respectively.

Shrinivasan et al[8] , Otasuka et al [9] and Abraham et al[10] have studied the reduction behavior of iron ore oxide-carbon mixture, where higher activation energy value during initial stage and gradual decrease with the progress of reaction were observed. The reaction has been proposed to be controlled by carbon gasification reaction, which is catalyzed by the presence of metallic iron. But in case of study made by Mukherjee et al[11] , the catalytic effect was because of a particular geometry of the sample. Due to this reason; the values of activation energies were 130.7, 152.1, 144.7 and 146.3 KJ/mole respectively. Mukherjee et al also reported the increase in reaction rate on addition of 5% Na_2CO_3 which is well known catalyst for carbon gasification reaction.

Bryk and Lu [12] studied the reduction behavior of commercial magnetite concentrate and carbon mixture in the temperature of 900-13000C. They have concluded that reaction kinetic was affected by furnace temperature, heat transfer, particle size of coal, coal: ore ratio and the reducing agent. 10

Coal and graphite have been reported to behave differently. Further replacement of coal by graphite has been reported to slow down the metallization process [12].

CHAPTER-3

EXPERIMENTAL

3. EXPERIMENTAL

3.1EXPERIMENTAL PROCEDURE

- Iron ore was collected from Sakaruddin mines of Orissa
- Coal was taken from lingaraj mines.
- Chemical analysis of the ore and proximate analysis of coal was done which is given in Table 3.11 & 3.12 respectively.
- Grinding of the ore to -100#, (-18+25) # and (-8+16) # was done.
- Crushing of coal to (-8+16) # was done.
- Making of pellets by hand rolling adding proper amount of water as binder.
- Pellets were made in size range of about 15 mm with the following variations in particle size.

Pellet type	Composition (% by wt)
A	-100# (100%)
B	{-100# (80%)} + {-18+25# (10%)} + {-8+16# (10%)}
C	{-100# (70%)} + {-18+25# (15%)} + {-8+16# (15%)}
D	{-100# (60%)} + {-18+25# (20%)} + {-8+16# (20%)}

- Air drying of the pellets followed by oven drying at 110^{o}C to remove moisture.
- Each type of pellet of above stated composition was fired at 900, 1000, 1100, 1200^{o}C for 1 hr to obtain proper strength.
- Separate crucible was taken for each type of pellet and marking was made for identification.
- Crushing strength of each type of pellet fired at 900 1000 1100 1200 ^{0}C was calculated which is given in Table 4.11.
- The apparent porosity values of different grade iron ore pellets were determined by using kerosene oil as a medium which is given in table.
- Reduction studies of fired pellets were carried out in a stainless steel container (size: 75mm height x 40mm inside diameter).
- The reactor was made full with the non coking coal of size (-8+16) #.

- Diameter (mm) of the fired pellet of the required grade was taken and weight(g) measurement was done and then it was placed centrally on this coal bed and the remaining portion of the stainless steel container was filled with coal so as to cover the fired pellet completely. The stainless steel container had a hole centrally for the escape of gas.
- The stainless steel container having coal and pellet was then introduced into the muffle furnace and heated from room temperature to the required temperature of 900ºC, 950ºC soaked there for 0, 15, 30, 60 minutes respectively.
- The containers were then taken out and cooled in air. The weight losses in pellets were recorded by an electronic balance and final diameter measurements were done with the help of vernier caliper .
- Calculation for degree of reductions and percentage swelling were made.

3.2DETERMINATION OF COLD CRUSHING STRENGTH

The crushing strength of fired pellets and some of reduced pellets (size 15mm) have been determined by employing a cold uniaxial hydraulic press (capacity 20 tons). The reported values of crushing strength were calculated by using the following formula .

$\sigma c = W/A$

Where, σc is the crushing strength in $kg.cm^{-2}$

W is the maximum load at fracture in kg

And A is the area in cm^2

3.3DETERMINATION OF APPARENT POROSITY

The apparent porosity values of iron ore lumps and pellets were determined by using kerosene oil as a medium in accordance with the following formula [12]

Apparent porosity = W – D/ W- (S-s)

Where, 'D' is the weight of dried piece;

'W' is the weight of oil saturated;

'S' is the weight of the piece + wire cage while immerse in oil;

and's' is the weight of wire cage only while immerse in oil

3.4DETERMINATION OF DEGREE OF REDUCTION

The degree of reduction was calculated by using the following formula:

Degree of reduction = (weight loss in pellet/total oxygen content in the pellet) ×100

3.5DETERMINATION OF PERCENTAGE SWELLING

Swelling is a volumetric expansion of the agglomerate during carbothermic reduction of iron oxide. Changes in crystal structure take place during the stepwise reduction of hematite through magnetite and wustite to metallic iron. These changes are accompanied with change in volume [13]. Percentage swelling can be calculated as:

$$\%\ Swelling = \frac{V_f - V_i}{V_i}$$

V_f = final volume of the reduced pellet

V_i= initial volume of the fired pellet

Swelling up to 20% has generally been accepted as ''normal'' whereas the high values are called ''abnormal swelling'' or even ''catastrophic swelling''

3.6PROCESS FLOW CHART

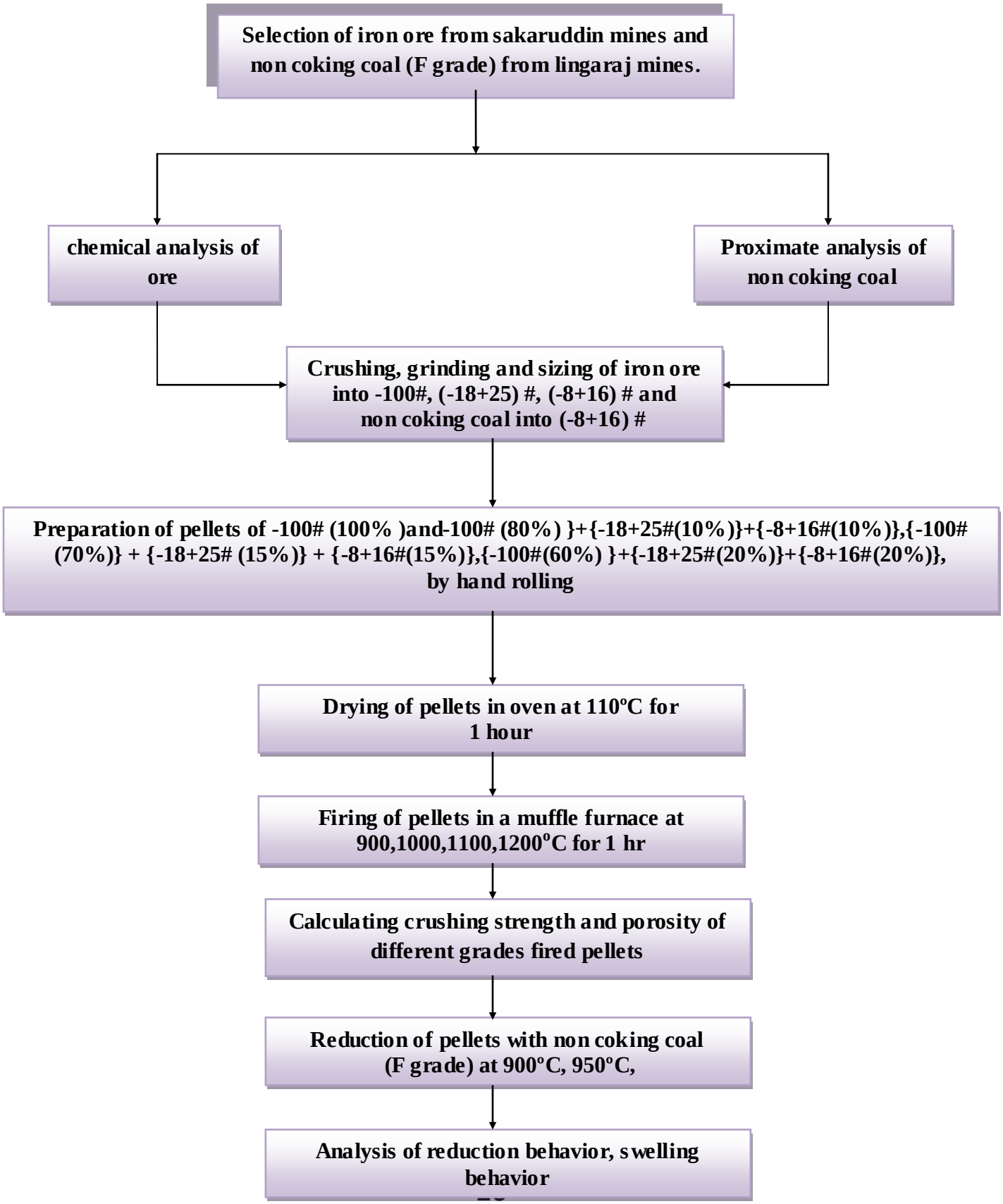


Table 3.11

Chemical analysis of iron ore [6]

Iron ore	Fe(total)	Fe_2O_3	Al_2O_3	SiO_2	TiO_2	MnO	Loss on ignition
Sakaruddin mine	64.51	91.74	3.06	1.43	0.14	0.02	3.61

Table 3.12

Proximate analysis of non coking coal*

Moisture	5%
Volatile matter	29%
Ash	22%
Fixed carbon	44%

*The reactivity of Lingaraj coal was found to be 4.19 cc/gm of CO/seconds.

CHAPTER-4

RESULTS & DISCUSSIONS

4. RESULTS AND DISCUSSIONS

4.1 EFFECTS OF FIRING CONDITION ON THE CRUSHING STRENGTH AND POROSITY OF THE PELLETS

In the present project work, pellets were fired at various temperatures in the range 900-1200^{0}C.for 1 hour. As shown in Table 4.11 and Figs. 4.1 & 4.2, the Crushing Strength of fired pellets increased with increase in firing temperature. As reported in the literature (Tupkary), this appears to be due to increase in the extent of slag bond in the pellets. The effect of firing temperature on the Crushing Strength is more pronounced at firing temperature of 1100^{0}C and 1200^{0}C. Table- 4.11and Fig 4.2 also clearly indicate that the Porosity of the fired pellets decrease wit increase in firing temperature. The decrease was found to be more pronounced at firing temperature of 1200^{0}C. This appears to be due to slag formation and filling of the pores at a firing temperature of 1200^{0}C.

4.2 EFFECT OF ADDITION OF +100 MESH SIZE PARTICLES ON THE CRUSHING STRENGTH AND THE POROSITY OF FIRED PELLETS

As shown in the Table 4.11 and Figs 4.1 & 4.2, The Crushing Strength and Porosity of the fired pellets were affected by the incorporation of +100 mesh size particles in the conventional pellets .The addition of +100 mesh size particles reduced the Crushing Strength and increased Porosity of resulting fired pellets.

The effect of addition of +100 mesh size particles on porosity is more pronounced at a firing temperature of 1200^{0}C.

4.3 EFFECT OF FIRING TEMPERATURE ON THE EXTENTS OF REDUCTION AND SWELLING FIRED PELLETS

As shown in Table 4.12, a decrease in degree of reduction was observed with increase in firing temperature. This appears to be due to lower porosity in the pellets fired at higher temperature (1200^{o}C). The extent of swelling during reduction also got drastically reduced with increase in firing temperature. This also appears to be related with the porosity and Crushing Strength of the pellets.

4.4 EFFECT OF REDUCTION TEMPERATURE ON THE DEGREE OF REDUCTION AND EXTENT OF SWELLING OF FIRED PELLETS

Data listed in Table 4.12 clearly indicate that degree of reduction increases and the extent of swelling decrease with increase of reduction temperature. An increase in the degree of reduction with reduction temperature appears to be more and more participation of gaseous reducing agents (CO and H_2) released from the devolatilization of coal and $\mathbf{C + O_2 \rightarrow} 2CO$ reaction.

A decrease in swelling index with increase in reduction temperature is more likely to be due to sintering of iron whiskers at higher temperature.

4.5 EFFECT OF REDUCTION TIME ON DEGREE OF REDUCTION AND EXTENT OF SWELLING OF FIRED PELLETS

The results shown in Table 4.12 and presented graphically in Fig.4.3, indicates that the degree of reduction increases with increase of time at a particular reduction temperature. This increase in degree is due to the exposure of pellets with the reducing agents (C, CO, and H_2) for a longer period of time .The higher reduction rate in initial conditions may be attributed to the combined effect of less resistance offered to the flow of reducing glass into the pellet and significance contribution of volatile matter release initially, as suggested by Bodsworth et al [14]. It is also clear from this table that the extent of swelling in the pellets fired at a temperature up to 1100^0C, in general, increases with increase of reduction time. This is believed due to fibrous growth of iron whiskers in the pellet matrix without any restriction in the growth; however, the pellets are fired at 1200^0C, in general, showed a decrease in the extent of swelling with rise of reduction time. This appears to be due to lower porosity in the pellets fired at 1200^0C. Lower porosity restricts the fibrous growth of whiskers and does not allow for expansion in the volume.

It may also be noticed that the effect of reduction time in increasing the degree of reduction is less pronounced than the effect of reduction temperature.

4.6 EFFECT OF ADDITION OF +100 MESH SIZE PARTICLES ON THE EXTENT OF SWELLING OF REDUCED PELLETS

As shown in Table 4.12, the addition of +100 mesh size particles in the matrix of -100 mesh size particles, in general, increases the extent of swelling in the reduced pellets. This is expected to be due to higher porosity and lower Crushing Strength.

As shown in the Table 4.12, the pellets made by the incorporation of +100 mesh size particles .Variation in the amounts of different sizes of +100 mesh size particles did not appear to have any significant effect on the extent of swelling.

4.7 SCANNING ELECTRON MICROGRAPHIC STUDY OF THE REDUCED PELLETS

The Scanning electron micrographic observations (Figs 4.9, 4.10) of the reduced pellets clearly indicate that the extent of sintering of iron particles in the pellets reduced at 900^0C and 950^oC. The SEM micrographs (Figs 4.11, 4.12) also indicate that the cracks formed in the pellets reduced at 950^0C are more pronounced than the cracks formed in the pellets reduced at 900^0C

4.8 EFFECT OF ADDITION OF CONCENTRATED SUGARCANE JUICE ON MECHANICAL PROPERTIES AND EXTENT OF REDUCTION OF FIRED PELLETS

Comparison of the data listed in Table 4.11, 4.12 and Table 4.13 clearly indicate that the addition of concentrated sugarcane juice in the pellets drastically increase the crushing strengths of resulting fired pellets. Not only this, the porosity of the resulting pellets also gets increased to some extent. An increase in crushing strength with the addition of sugar juice as a binder is believed to be due to the developments of strong binding force between the iron ore particles. The degree of reduction in the resulting pellets has got increased to some extent. The reason for this is yet to be found out.

Table 4.11

Results obtained for the Mechanical Properties of fired Pellets

Sample	Firing conditions			
Pellet type *	Temperature(^{o}C)	Time (minutes)	Crushing strength(kg)	Porosity
A	900	60	50	28.3
A	1000	60	50	27.7
A	1100	60	60	26.84
A	1200	60	65	16
B	900	60	30	25
B	1000	60	50	25.2
B	1100	60	50	25.6
B	1200	60	50	18
C	900	60	20	-
C	1000	60	45	-
C	1100	60	45	26
C	1200	60	45	19
D	900	60	20	-
D	1000	60	20	-
D	1100	60	35	22.5
D	1200	60	40	21.6

*A [-100# (100%)],

B [{-100# (80%)} + {-18+25# (10%)} + {-8+16# (10%)}]

C [{-100# (70%)} + {-18+25# (15%)} + {-8+16# (15%)}]

D [{-100# (60%)} + {-18+25# (20%)} + {-8+16# (20%)}]

Table 4.12

Results obtained for the extents of reduction and swelling of fired pellets

Sample	Reduction conditions				
Pellet type	Firing temperature(^{o}C)	Reduction temperature(^{o}C)	Reduction time(minutes)	%Reduction	%Swelling
A	1200	900	15	95.59	5.64
A	1200	950	15	100	4.6
A	1100	900	30	96.37	8
A	1100	900	60	100	4.35
B	1100	900	0	84	20
B	1100	900	30	87.93	24
B	1100	900	60	100	8
B	1200	900	15	95.29	42.47
B	1200	900	30	97.43	6.537
B	1200	950	0	85.303	6.9
B	1200	950	15	95.01	22.9
C	1000	900	60	100	4.3
C	1100	900	30	98.53	3.03
C	1100	900	60	100	3.22
C	1200	900	15	95.68	31.82
C	1200	950	15	96.22	25.7
D	1000	900	60	100	-2.33
D	1100	900	30	95.45	2.86
D	1100	900	60	100	-3.811
D	1200	900	15	97.07	10.88
D	1200	950	15	100	-2.56

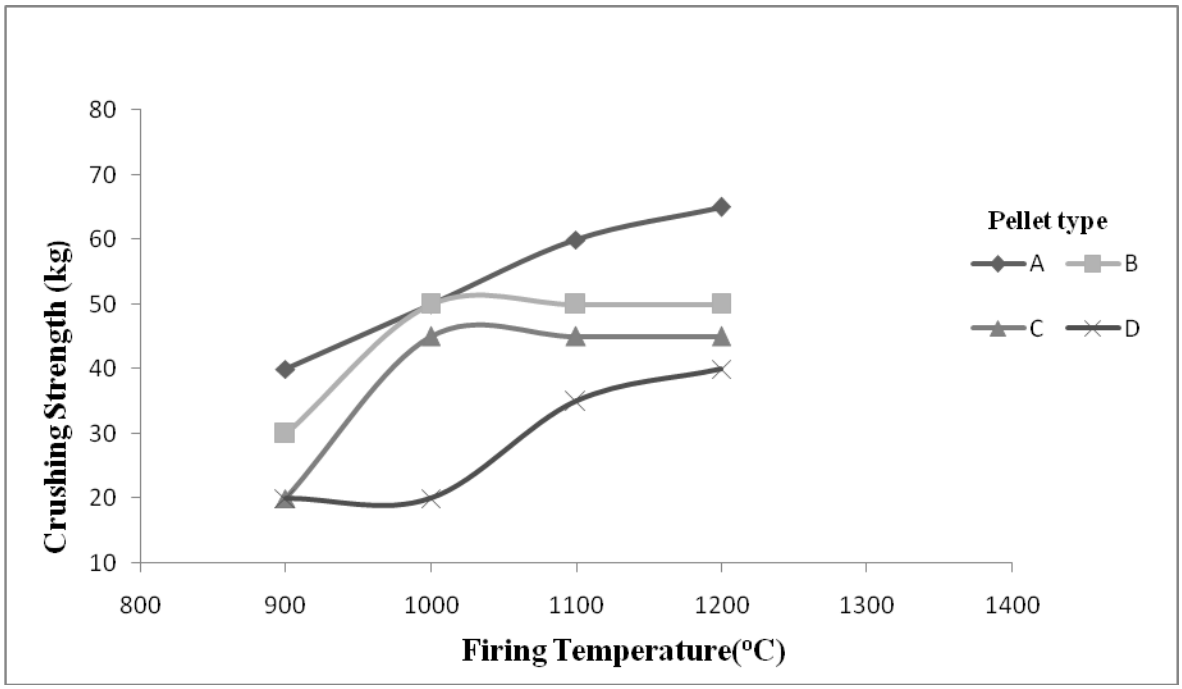


Fig: 4.1 Crushing Strength (kg) Vs Firing Temperature (^{o}C) for different types (A, B, C, D) of iron ore pellet

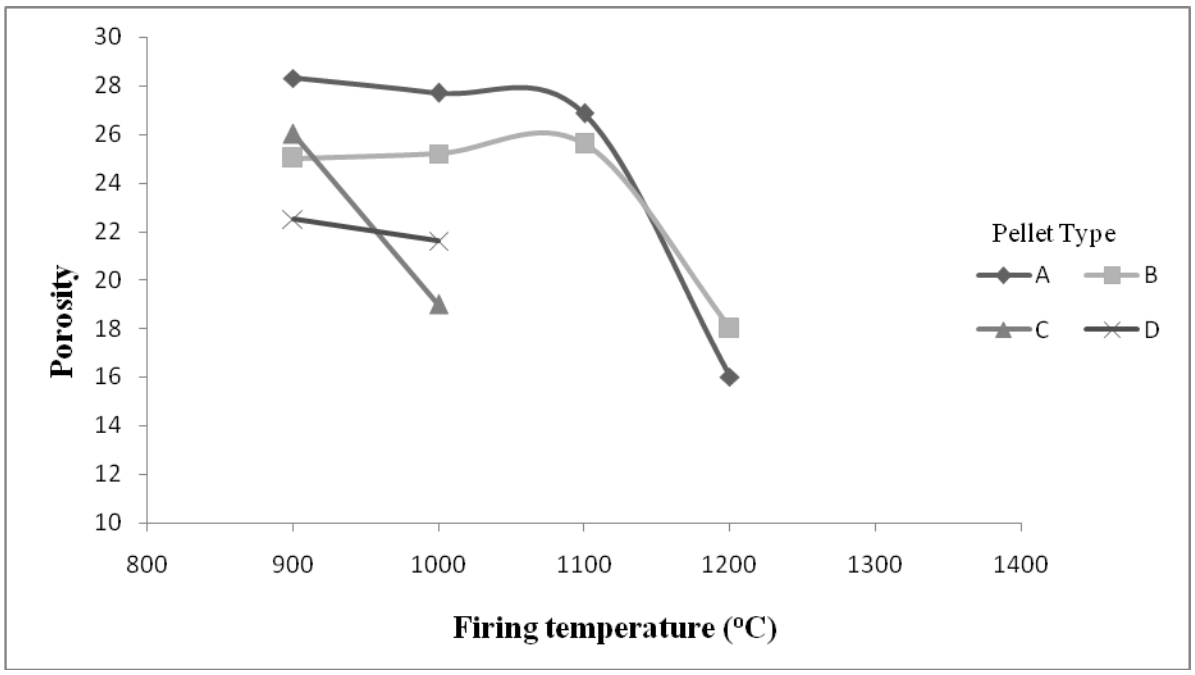


Fig: 4.2 Porosity Vs Firing Temperature (^{o}C) for different types (A, B, C, D) iron ore pellet

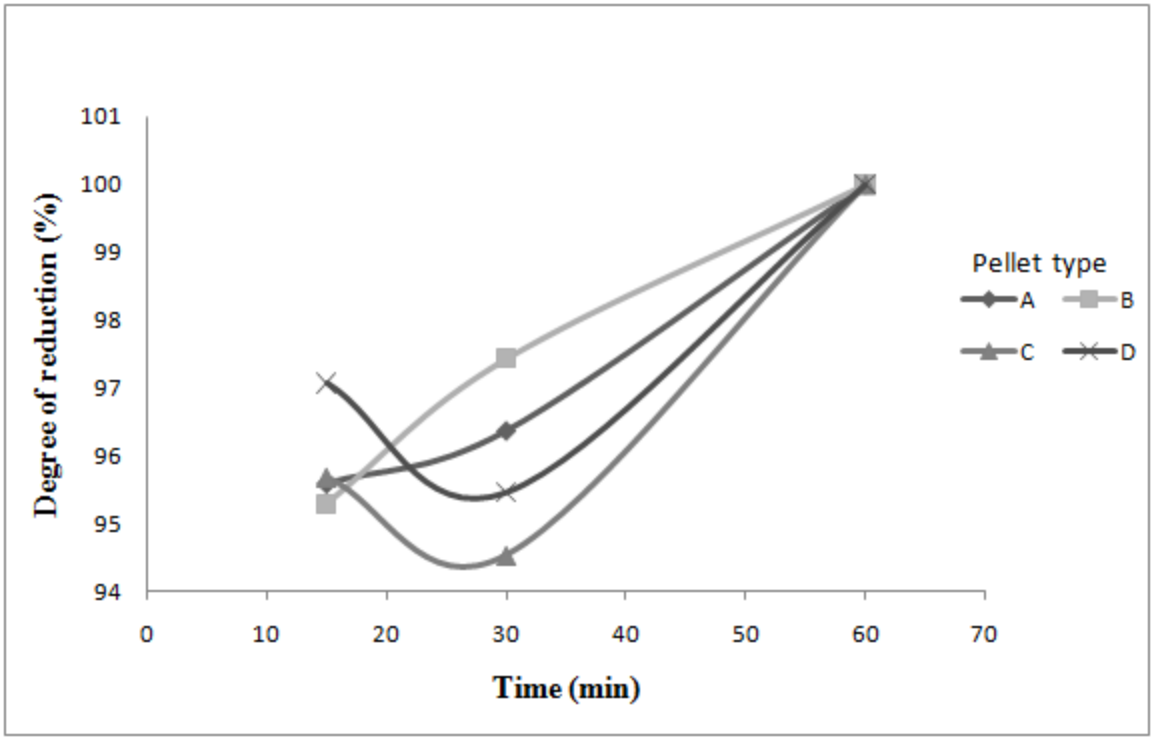


Fig: 4.3 Degree of reduction (%) Vs Time (min) for different types (A, B, C, D) of iron ore pellet

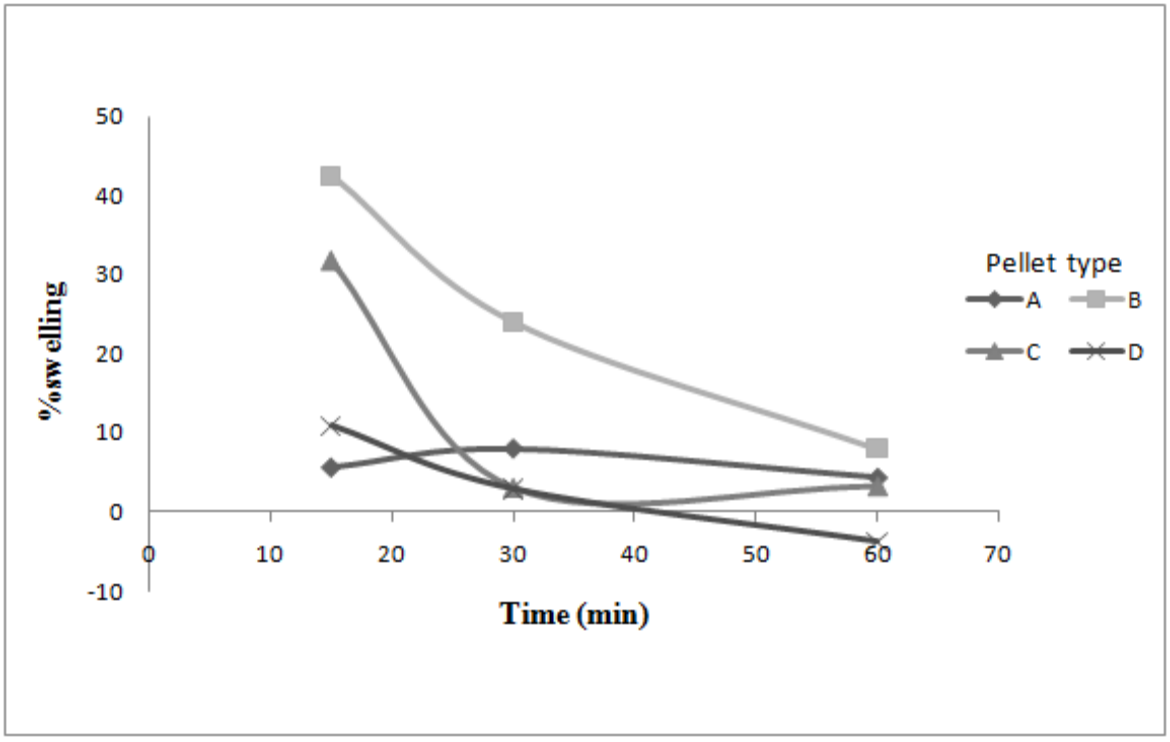


Fig: 4.4 Swelling (%) Vs Time (min) for different types (A, B, C, D) of iron ore pellet.

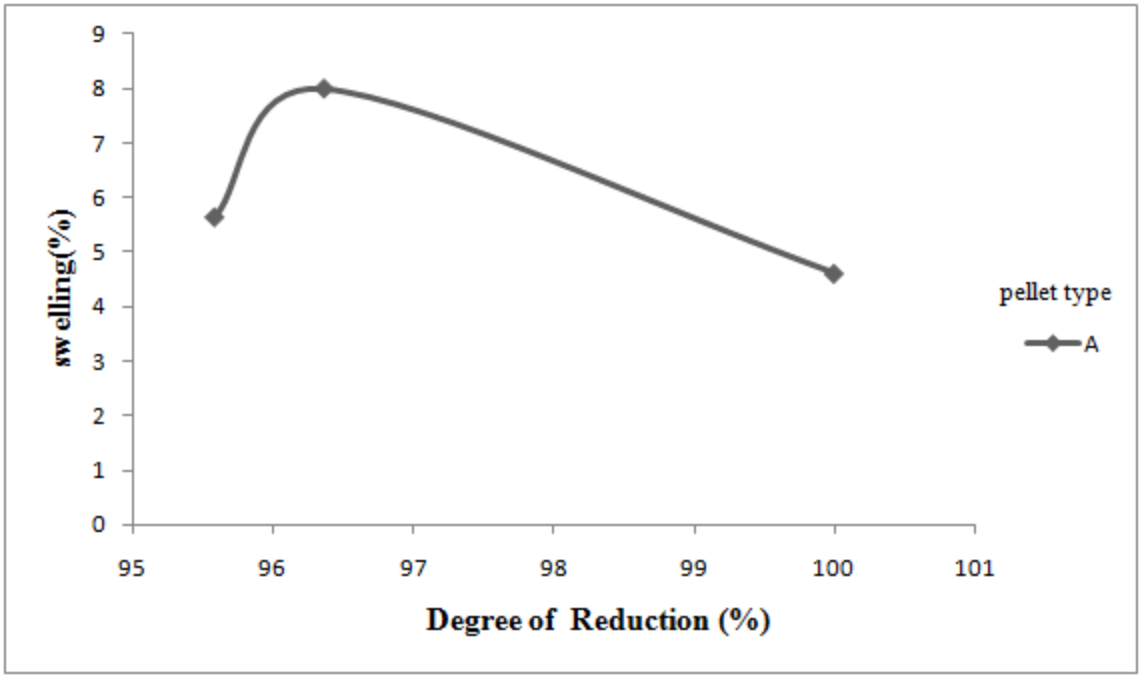


Fig: 4.5 Swelling (%) Vs Degree of Reduction(%) for A type {-100# (100%)} of iron ore pellet

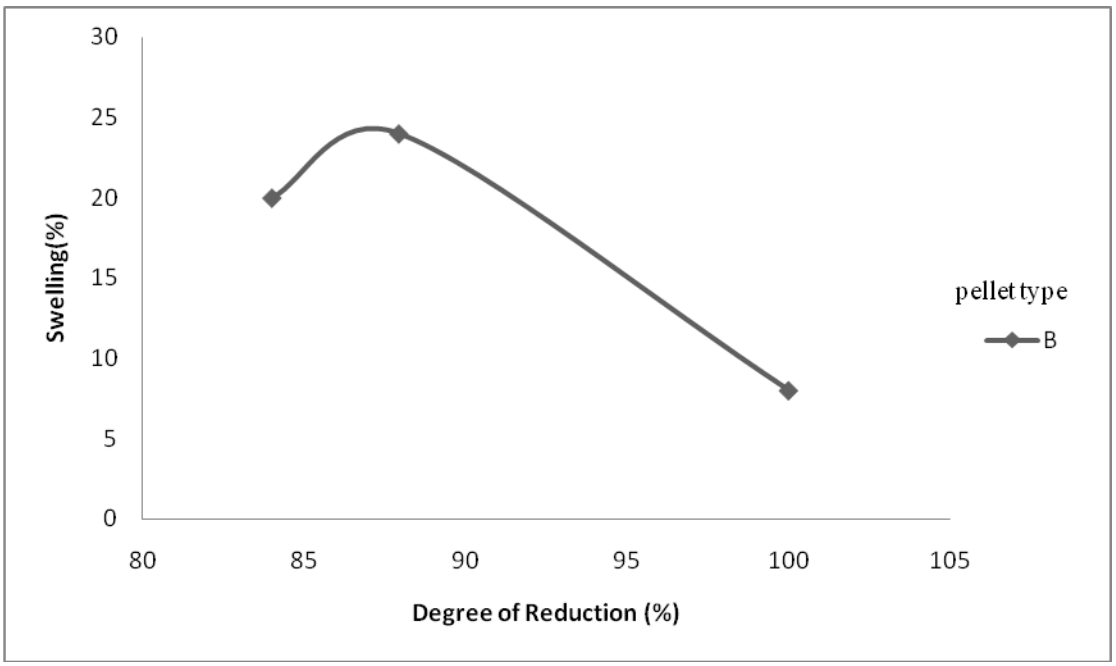


Fig: 4.6 Swelling (%) Vs Degree of Reduction(%) for B type [{-100# (80%)} + {-18+25# (10%)} + {-8+16# (10%)}] of iron ore pellet

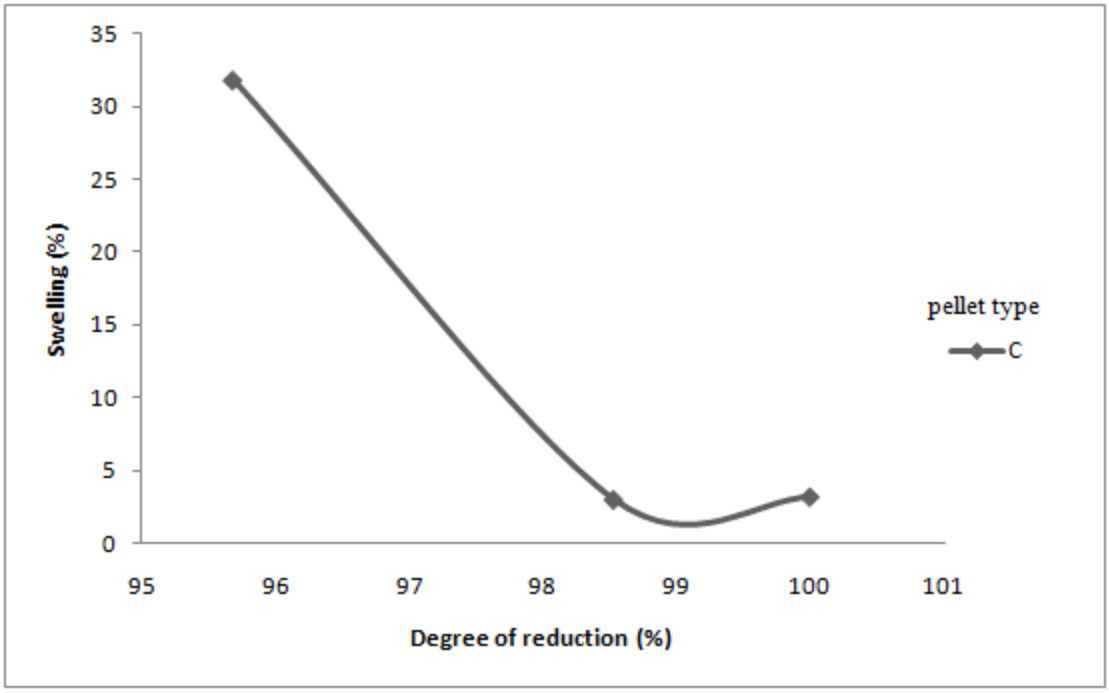


Fig: 4.7 Swelling (%) Vs Degree of Reduction(%) for C type [{-100# (70%)} + {-18+25# (15%)} + {-8+16# (15%)}] of iron ore pellet

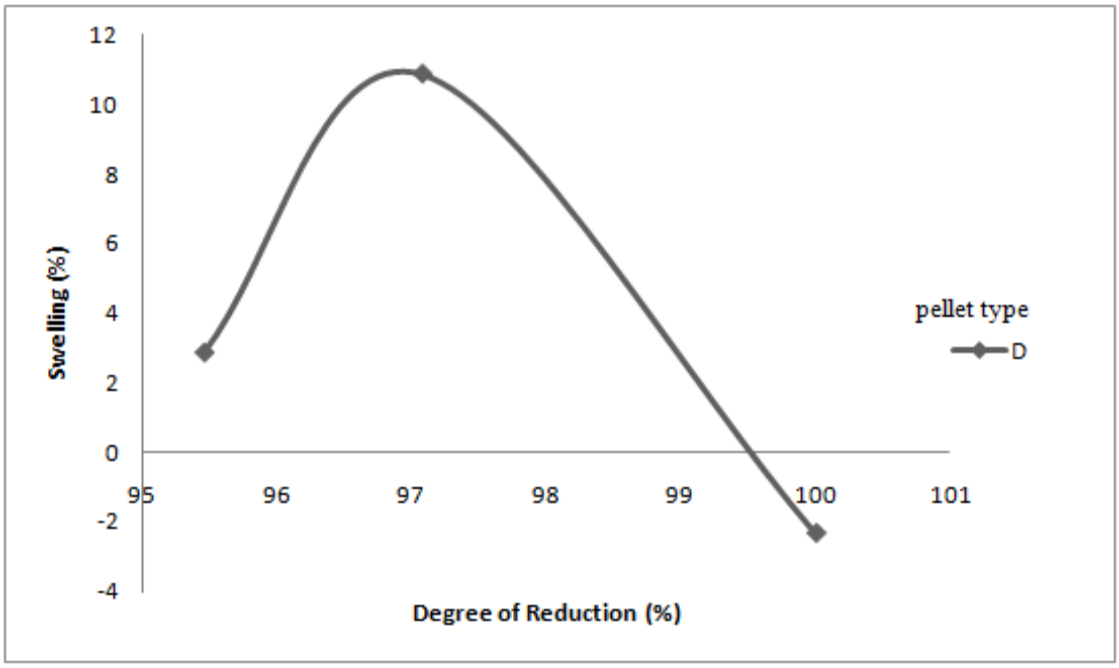


Fig: 4.8 Swelling (%) Vs Degree of Reduction for D type [{-100# (60%)} + {-18+25# (20%)} + {-8+16# (20%)}] of iron ore pellet

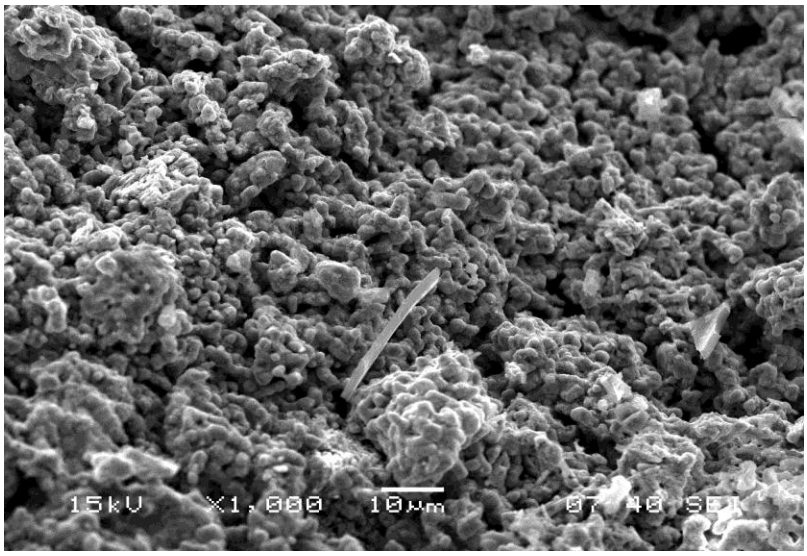


Fig: 4.9 SEM image of Sakaruddin iron ore pellet B type [{-100# (80%)} + {-18+25# (10%)} + {-8+16# (10%)}] reduced at temperature 900^{o}C, Magnification 1000X.

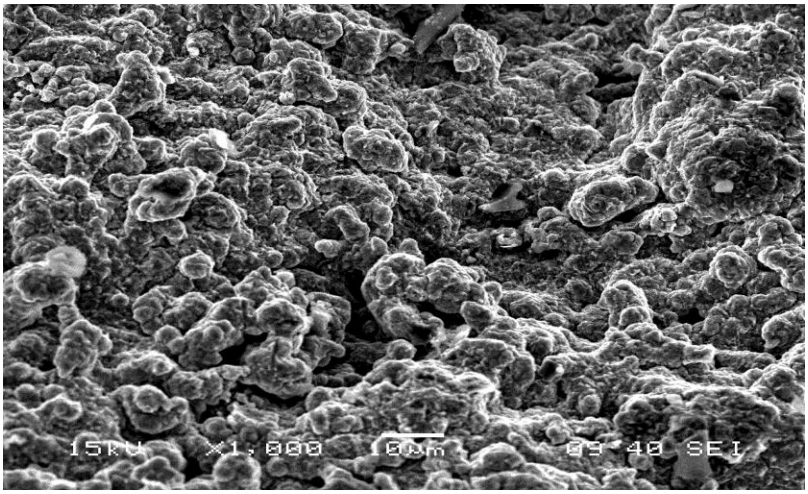


Fig: 4.10 SEM image of Sakaruddin iron ore pellet A type {-100# (100%)} reduced at temperature 950^{o}C, Magnification 1000X.

Fig: 4.11 SEM image of Sakaruddin iron ore pellet A type {-100# (100%)} reduced at temperature 950^{o}C, Magnification 100X.

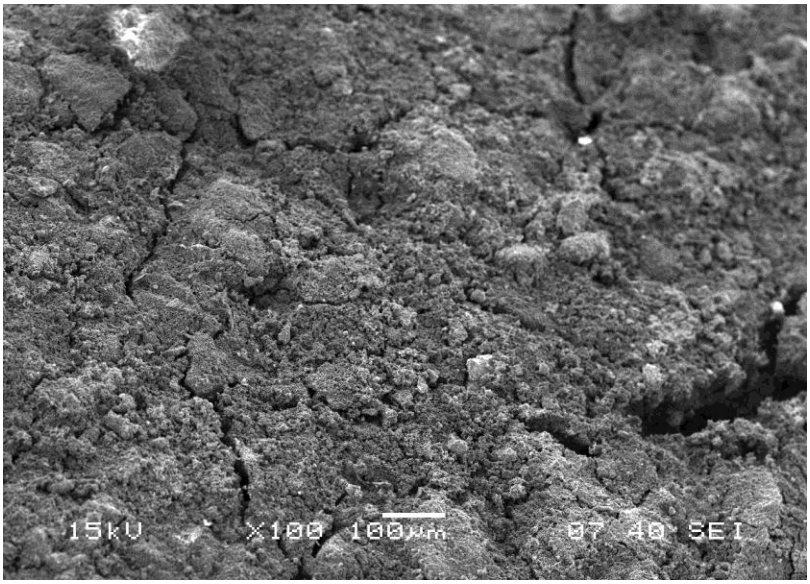


Fig: 4.12 SEM image of Sakaruddin iron ore pellet B type [{-100# (80%)} + {-18+25# (10%)} + {-8+16# (10%)}] reduced at temperature 900^{o}C , Magnification 100X.

Table 4.13

Results obtained for the extent of reduction and swelling of fired pellets made by the addition of concentrated sugar cane juice (10% by wt)

Sample	Firing condition	Mechanical properties		Reduction conditions			
Pellet Constituent {-100# (80%)} + {-18+25# (10%)} + {-8+16#(10%)}	Firing-Temperature 1100^{o}C Firing time-60 minutes	Crushing strength (kg)	Porosity	Reduction temperature (^{o}C)	Reduction time (minutes)	Degree of reduction (%)	Swelling (%)
Sample I	do	110	25.4	900	60	100	6
Sample II	do	100	22.5	900	60	100	4.8

CHAPTER-5

CONCLUSIONS

5. CONCLUSIONS

- The Crushing Strength of fired pellets increased with increase in firing temperature. The effect of firing temperature on the Crushing Strength is more pronounced at firing temperature of 1100^0C and 1200^0C.
- Porosity of the fired pellets decrease with increase in firing temperature. The decrease was found to be more pronounced at firing temperature of 1200^0C.
- The addition of +100 mesh size particles reduced the Crushing Strength and increased Porosity of resulting fired pellets.
- A decrease in degree of reduction was observed with increase in firing temperature from 900^oC to 1200^oC.
- Degree of reduction increases and the extent of swelling decrease with increase of reduction temperature from 900 to 950^oC.
- The degree of reduction increases with increase of time at a particular reduction temperature.
- The extent of swelling in the pellets fired at a temperature up to 1100^0C, in general, increases with increase of reduction time.

- The reduction behavior of mixed iron ore pellets of different composition (-100#) 80% + (-18+25#) 10% + (-10+16#) 10%; (-100#) 70% + (-18+25#) 15% + (-10+16#) 15%;(-100#) 60% + (-18+25#) 20% + (-10+16#) 20% were found to be identical with the iron ore pellets made by -100# iron ore fines. This is of greatest advantage in the use of these iron ore pellets in rotary kiln for sponge iron production, which can lead to a saving of enormous amount of energy.
- The additions of concentrated sugarcane juice in the pellets drastically increase the crushing strengths of resulting fired pellets. Not only this, the porosity of the resulting pellets also gets increased to some extent.

CHAPTER-6

FUTURE WORK

6. SUGGESTIONS FOR THE FUTURE WORK

The works carried out in this area may be extended in the future by other investigators. The suggested future work is as follows-

1. Detailed study on the reduction kinetics of fired pellets made by the addition of +100 mesh size particles may be carried out.
2. Necessary steps need to be taken to improve the crushing strength of the fired pellets.
3. Effects of the addition of different types of binders on the crushing strength and the reduction kinetics of the non conventional fired pellets may be studied.

CHAPTER-7

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